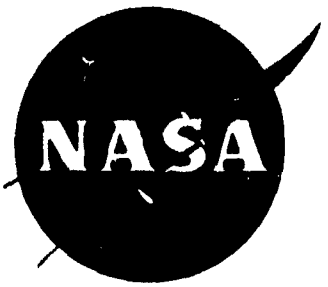

TECHNICAL NOTE

D-699

A SPECTROPHOTOMETRIC ATTACHMENT FOR THE VACUUM ULTRAVIOLET

Norman N. Axelrod

Goddard Space Flight Center
Greenbelt, Maryland

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SUMMARY

An absorption spectrophotometric attachment to a vacuum ultraviolet monochromator has been built and tested. With the sample chamber empty, the ratio of the radiant flux through the sample chamber to the radiant flux through the reference chamber was measured. By optimizing the conditions at the entrance slit, the ratio was kept constant within experimental error over the region 1000 to 1600A. The transmittance of thin celluloid films was measured with the attachment.

CONTENTS

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INTRODUCTION

In order to facilitate absorption measurements in the vacuum ultraviolet, a monochromator attachment has been built and tested, which makes use of the top and bottom halves of the exit beam of a vacuum monochromator. Light pipes with phosphor-covered ends are placed in front of each half of the exit slit, and the phosphorescence is piped to two separate photomultiplier tubes. Small samples are introduced in front of the phosphor-covered end of one light pipe, and the other pipe provides a reference source. The two signals can then be read simultaneously on two pen recorders, or the outputs of the amplifiers can be fed into a ratio recorder. Although sodium salicylate (References 1 and 2) was used in this instance, it is apparent that other phosphors (Reference 3) can be used and the ratio of the outputs of the two photomultipliers, with no sample in either half of the beam, would remain constant as a function of wavelength.

DESCRIPTION OF THE APPARATUS

The possibility of crosstalk, once the radiation is beyond the sample holder, is eliminated by means of mechanical obstructions (Figure 1). The light pipes were made of Pyrex, coated with evaporated aluminum, and sprayed with transparent paint for durability. The photomultiplier tubes were E. M. I. 9256B's, operated at 940 volts. The only vacuum seals necessary were the Glyptal seals around the light pipes and the O-ring seals on the face plate and on the plunger that holds the samples. The flat part of the sample holder was milled so that it had a clearance of 0.005 inches from the metal in front of the light pipes. The vertical position of the samples was adjusted by a hole-and-pin arrangement in the plunger, and the azimuth was fixed by the milled sample holder.

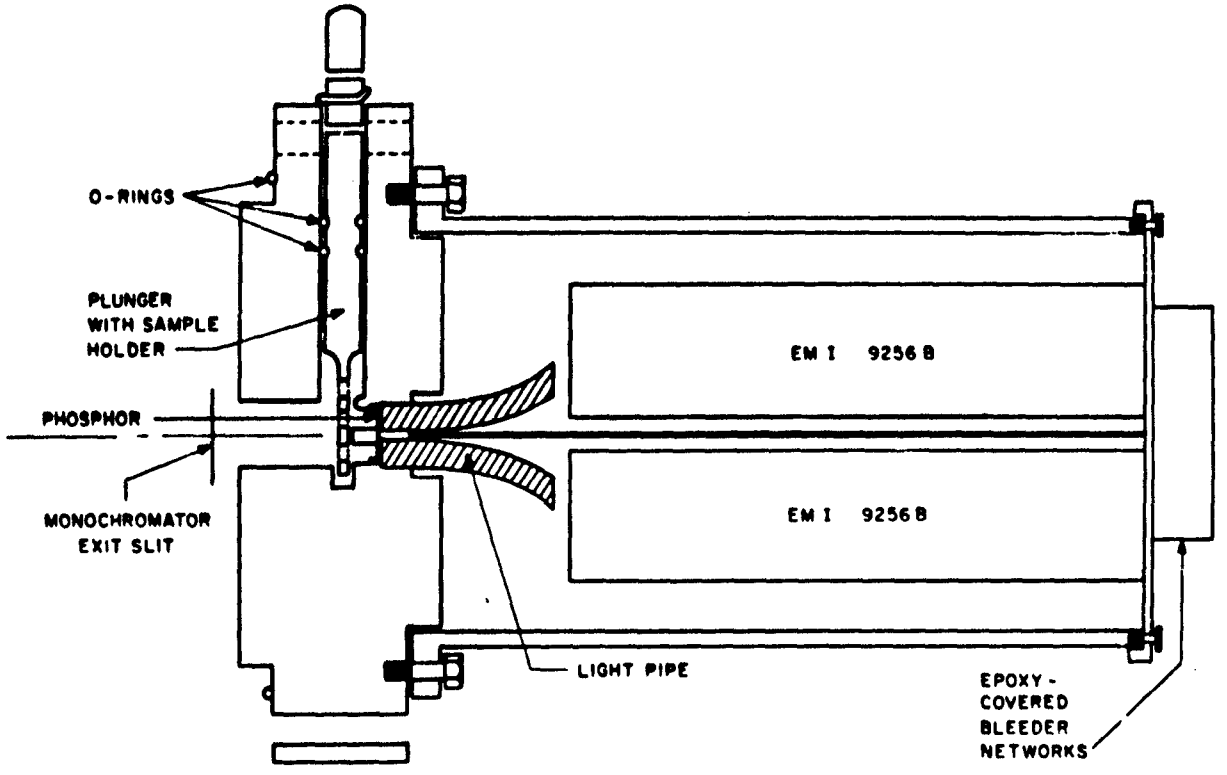

Figure 1 – The spectrophotometric attachment

The monochromator used was a McPherson No. 220 normal-incidence vacuum monochromator, which has a mount 1 meter in radius. The grating was an aluminum-coated replica with 600 lines/mm; with 100-micron entrance and exit slits, the measured full width of a line at half maximum was 2.6A. The operating pressure in the main chamber was less than 10^{-4} mm Hg. Simultaneous recordings of the phototube outputs were made on two Brown recorders.

DISCUSSION OF RESULTS

The first tests were made using a continuously operated 0.25-amp dc discharge through a water-cooled capillary (Reference 4). The fluxes were measured by scanning a line and using the peak value minus the minimum value. The ratio R of the signal through the bottom part of the slit to that through the top part was not constant as a function of wavelength. In fact, R varied over a range from 0.4 to 0.7 in a reproducible fashion. When the attachment was rotated 180 degrees about the center of the slit, the ratio R still varied in the same manner and had approximately the same values. This indicated that the variation was not inherent in the attachment, but rather was due to different spectral distributions at the top and the bottom of the slit.

To explain this nonconstancy of R, two possibilities seemed reasonable. The first was that the spectral distribution of the source varied as a function of position within the source; this, inasmuch as the image at the exit slit was stigmatic, would be sufficient to explain the variation of R. The other possibility was that each segment of the entrance slit was not illuminating the grating uniformly. If the reflectivity of different parts of the grating varied, then the stigmatic formation of the image would cause the spectral distribution along the exit slit to vary also. In Figure 2 can be seen the effect of the vignetting by the capillary tube. All the radiation passing through point A on the entrance slit from a plane normal to OO' and containing points B and C in the capillary tube is incident on the grating between P and Q. Since the image of the entrance slit is approximately focused on the exit slit, and hence on the detector, the spectral distribution from A on the detector will heavily weight the response of the top half of the grating. The front of the capillary tube was approximately 1.5 cm from the slit. The capillary had an inner diameter of 4 mm and was 9.5 cm long, while the entrance and exit slits were 12 mm long and 100 microns wide.

A more remote possibility (suggested by Dr. William E. Behring), which might cause a systematic error in monochromators with wide tolerances, is the movement of the image of the entrance slit by vertical motion of the grating. This would cause difficulty particularly if the image does not entirely cover both light pipes.

In order to reduce the variation in R, a new source with a different geometry was constructed and mounted in front of the entrance slit of the monochromator. This source was a cylindrical Pyrex tube of inner diameter 2.2 cm, with an aluminum tube suspended

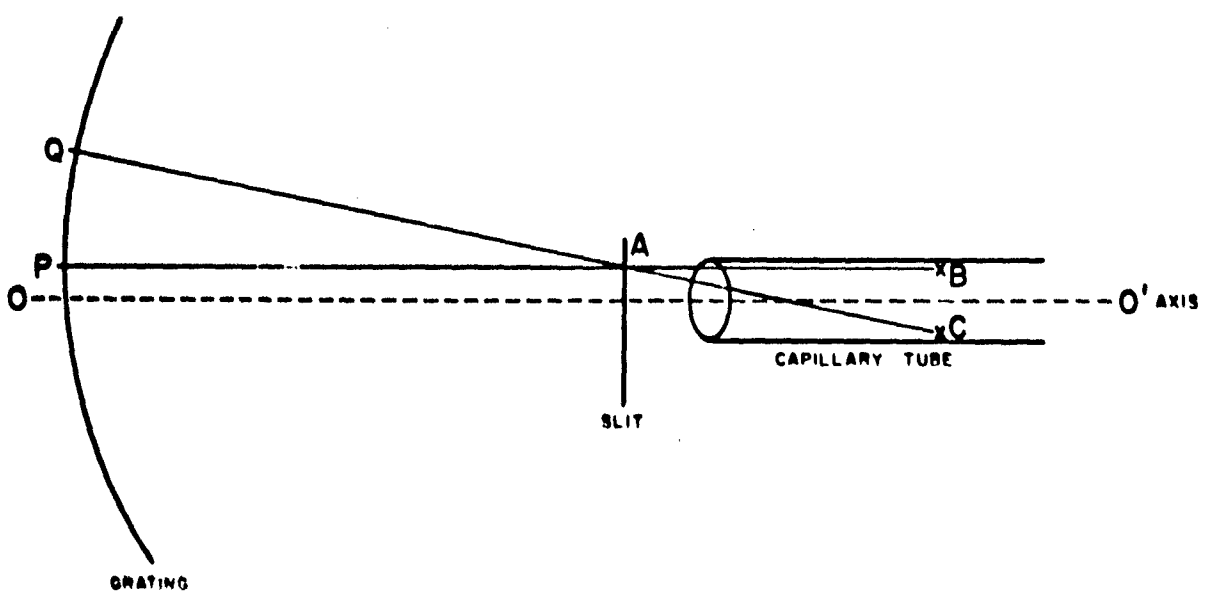

Figure 2 – Effect of vignetting by the capillary tube

along the same axis. This aluminum cylinder, suspended from the Pyrex tube by a tungsten wire, was 1 cm in diameter, 2 cm in length, and 2.5 cm from the aperture. The Pyrex tube was mounted with an O-ring on a grooved aluminum plate in front of the slit. The circular aperture in the plate had a diameter of 9 mm and was located 1.25 cm from the entrance slit. A nitrogen discharge, at pressures on the order of 100 microns Hg, was powered by a 125-watt 2450-mc Raytheon diathermy unit. It is probable, with this alteration, that the light going through the entrance slit was not as well collimated as it had been with the dc discharge tube, and that the effects of the wall were reduced. This arrangement made it possible to reduce the measured variations in R to less than 3 percent; however, it should be noted that the smaller variations observed when the diathermy-unit-power source was used had the same shape as the variations observed with the discharge tube. The gas used as the source did not appear to alter the dependence of R on wavelength.

The variation of the ratio R with wavelength for the discharge tube with free-flowing hydrogen at 350 microns Hg is shown as curve a of Figure 3. The best results obtained

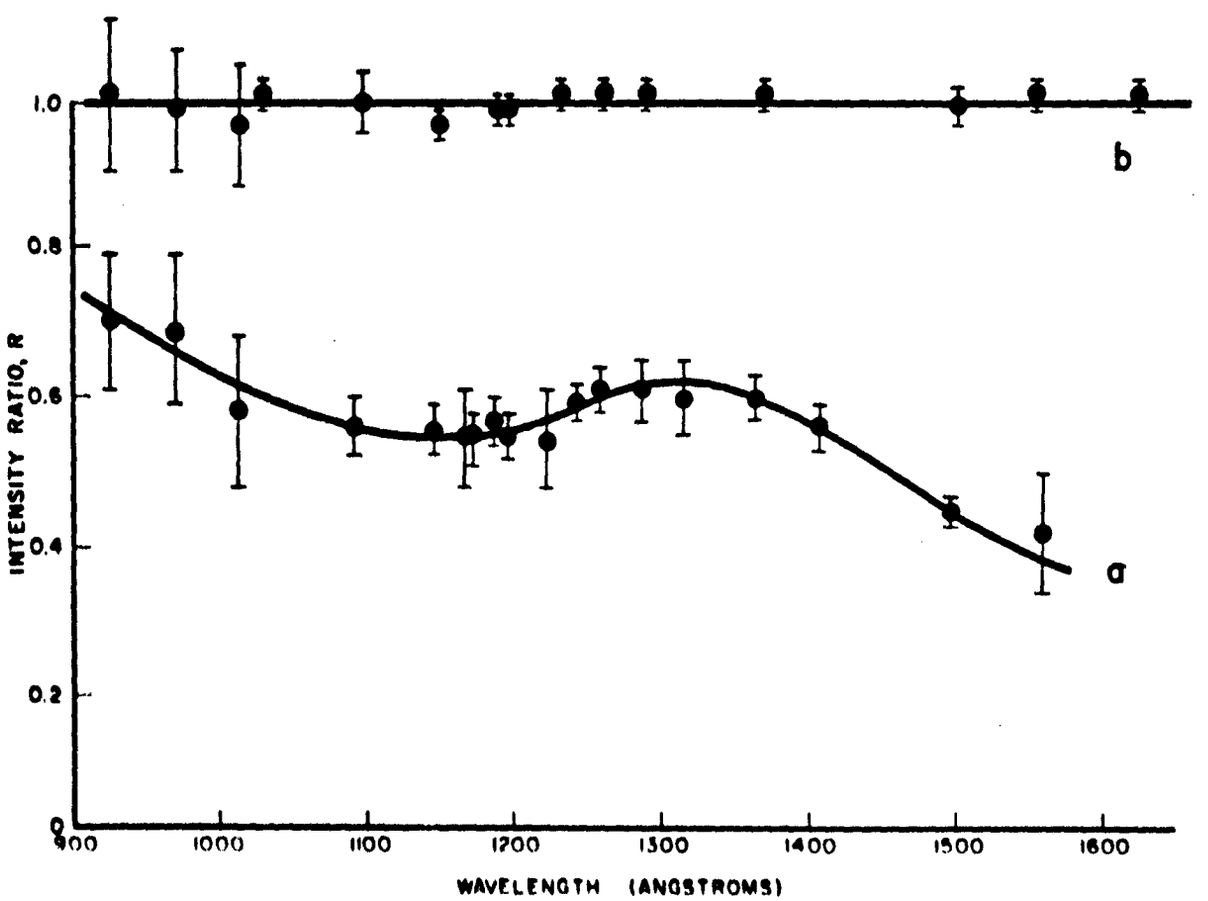

Figure 3 – The ratio R of the radiant flux through the sample chamber to the radiant flux through the reference chamber, as a function of wavelength: curve a for the dc discharge through the capillary; curve b for the 2450-mc discharge in 22-mm tubing

by using the microwave-powered source with hydrogen at 400 microns Hg are shown as curve b. The best microwave-tube results were obtained with aluminum foil shielding on the Pyrex tube with the region between the aluminum plate and the mouth of the aluminum tube uncovered. The foil confined the discharge to a region near the slit and quieted the photomultiplier signal.

In Figure 4, curves a and b are the product of the linear absorption coefficients of thin films of collodion and the film thickness as a function of wavelength. The collodion films were prepared by dissolving aged collodion in isoamyl acetate. The films, formed by placing a drop of the solution on the surface of distilled water, were lifted by a 300-mesh screen with 39 percent transmittance between 1000 and 1600A. The transmittance of the films was measured with no correction for the reflectance. The films used in this case appear to be the same as those prepared by H. M. O'Bryan (Reference 5). By employing his constants and Fresnel's equations, the reflectance at 1000A is found to be approximately 10 percent.

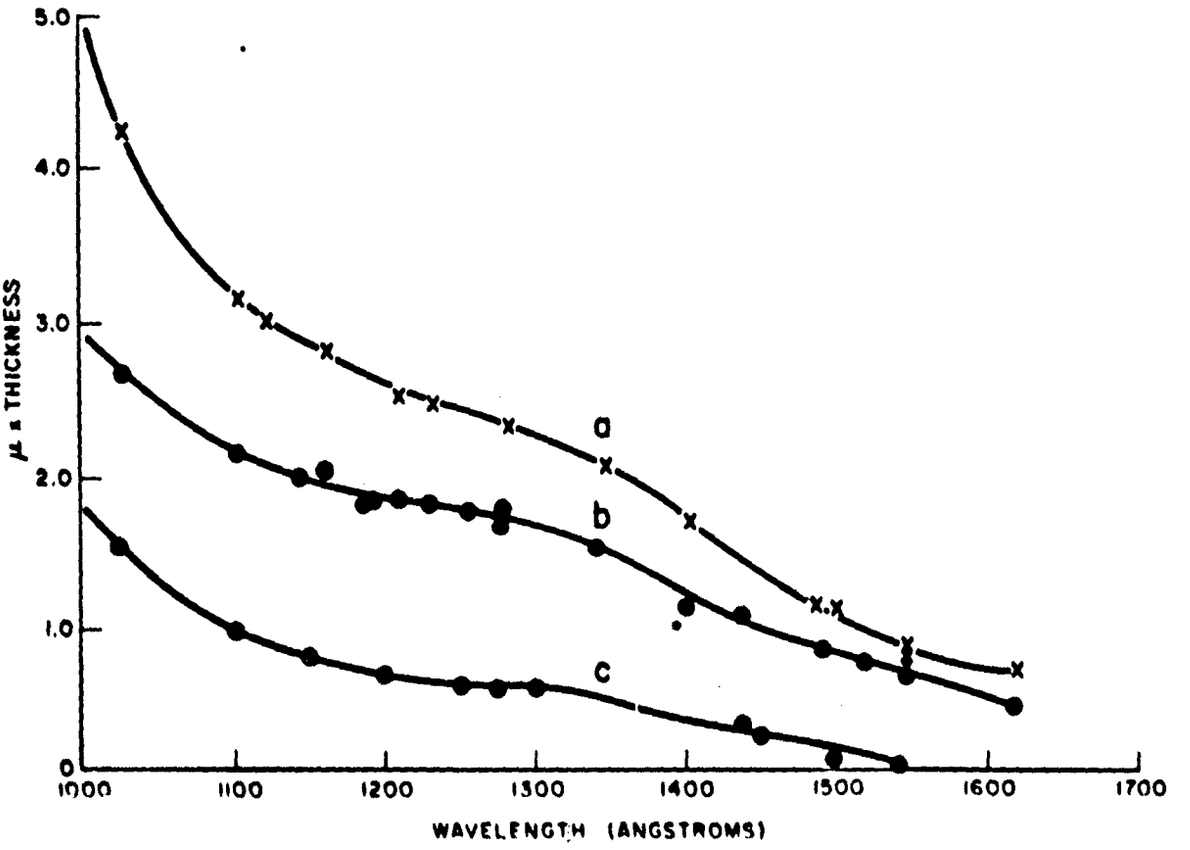

Figure 4 – Linear absorption coefficient times thickness as a function of wavelength. curve a for a collodion film 480 Angstroms thick; curve b for a collodion film 290 Angstroms thick. Curve c the difference between curves b and a.

Since the absorption coefficient is varying smoothly, the reflectance can also be expected to change in a regular manner. In curve c of Figure 4, the linear absorption coefficient obtained by subtracting curve b from curve a is seen to have the same shape as a and b. If there were a systematic error which was approximately the same in a and b, it can be shown by a simple calculation that the subtraction process would have removed the systematic error in much the same fashion as it would have removed the effect of the reflectance. Since these curves are identical within experimental error, the systematic error caused by the spectrophotometer is seen to be negligible. It is of interest to note that by extrapolating the curves a and b to 1000A and using O'Bryan's absorption coefficient, the thickness of sample a is found to be 480A, and that of sample b 290A.

EXTENSIONS AND SUGGESTIONS

For use with a ratio recorder, it is strongly advised that the image of the entrance slit be made parallel with the exit slit. This is considerably more important for a source with rapidly varying spectral distribution than in the case of a slowly varying continuum.

If it is considered necessary to reduce the dark current and noise of the photomultipliers, light pipes using fiber bundles could be used to transmit the luminescence over longer distances, and the tubes could conveniently be mounted in suitable cryostats.

A possible error with this spectrophotometric attachment would be the reflection of luminescence from the sample back into the light pipe. This problem could easily be resolved by tilting the sample so that the reflected light is directed onto a black surface or into a light trap.

ACKNOWLEDGMENTS

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